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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 150

RAW, REFINED AND BOILED LINSEED OIL

1st EDITION

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 150, *Raw, Refined and Boiled Linseed Oil*, was drawn up by Technical Committee ISO/TC 35, *Paints, Varnishes and Related Products and their Raw Materials*, the Secretariat of which is held by the Nederlands Normalisatie-instituut (NNI).

Work on this question by the Technical Committee began in 1950 and led, in 1958, to the adoption of a Draft ISO Recommendation.

In June 1958, this Draft ISO Recommendation (No. 158) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Austria
Burma
Chile
Czechoslovakia
Denmark
Germany
Greece
India
Ireland
Israel
Italy
Japan
Mexico
Netherlands
New Zealand
Poland
Portugal
Republic of South Africa
Romania
Spain
Sweden
U.S.A.
U.S.S.R.

Four Member Bodies opposed the approval of the Draft:

Belgium
Canada
France
United Kingdom

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1960, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 150 February 1960

RAW, REFINED AND BOILED LINSEED OIL

1. SCOPE

This ISO Recommendation establishes the more important requirements for raw, refined and boiled linseed oils and the methods of test for these requirements.

2. DEFINITIONS

2.1 *Raw linseed oil.* The oil obtained solely from mature seeds of linseed (*Linum usitatissimum* L.).

2.2 *Refined linseed oil.* The oil obtained by refining raw linseed oil as defined in clause 2.1.

2.3 *Boiled linseed oil.* The oil obtained from raw or refined linseed oil as defined in clause 2.1 or 2.2, by the incorporation of driers and by heating with or without blowing with air or oxygen.

3. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

The raw, refined and boiled linseed oils should have the following characteristics:

Property	Raw linseed oil	Refined linseed oil	Boiled linseed oil	Clause describing test method
Density (1) g/ml ρ_{20} (ρ_{40})	0.926 to 0.933 (0.912 to 0.919)	0.926 to 0.933 (0.912 to 0.919)	0.928 to 0.950 (0.914 to 0.936)	5.1
Colour	Not darker than test solution	To be agreed between purchaser and vendor	To be agreed between purchaser and vendor	5.2
Clarity	—	No sediment (2)	No sediment (2)	5.3
Refractive index (3) n^{20}_{D} (n^{40}_{D})	1.4790 to 1.4840 (1.4720 to 1.4770)	1.4785 to 1.4825 (1.4715 to 1.4755)	— —	5.4
Volatile matter, max. %	0.20	0.10	0.20	5.5
Ash, max. %	0.15	0.02 (4)	(5)	5.6
Acid value, max.	4	acid refined 9 (6)	8 (4)	5.7
Saponification value	188 to 195	188 to 195	185 to 200	5.8
Unsaponifiable matter, max. %	1.8	1.5	2.0	5.9
Iodine value, min. (Wijs method) (7)	175	175	—	5.10
Foots, max. %	1.0	nil	—	5.11
Drying time, max.	—	—	24 h at 15 to 20 °C or 18 h at 25 to 30 °C	5.12
Colophony	negative test	negative test	negative test	5.13
Fish oil	negative test	negative test	negative text	5.14
Mineral acid	—	negative test	—	5.15

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(1) The figures between brackets are for use in countries with a tropical climate.
As factor of variation per degree C, 0.000 68 is used.

(2) Stricter requirements may be agreed between purchaser and vendor. See also " Foots " and 5.11.

(3) The figures between brackets are for use in countries with a tropical climate.
As factor of variation per degree C, 0.000 35 is used.

(4) Or to be agreed between purchaser and vendor.

(5) It is recommended that requirements for the ash content be agreed between purchaser and vendor.
The ash may contain metallic oxides.

(6) The value for alkali refined linseed oil to be agreed between purchaser and vendor.

(7) Raw or refined linseed oil with an iodine value of over 190 should be designated " high iodine value linseed oil ".
If the Hanus method is used, it should be taken into account that this method can give different values from those obtained by the Wijs method.

4. SAMPLING

See ISO Recommendation R …, *Sampling Raw Materials for Paints and Varnishes* *.

5. TEST METHODS

5.1 Density

Refer to the Annex A, subject to the provision that ISO recommends that densities, like refractive indices, be quoted at 20 °C (and if necessary at 40 °C in countries with a tropical climate).

5.2 Colour

Prepare a solution of 0.08 g of iodine and 0.8 g of pure potassium iodide (free from iodate) in 100 ml of water. Compare the iodine solution with the oil in identical glass tubes, 1 cm in diameter and about 10 cm long, by viewing them transversely by transmitted light.

5.3 Clarity

The oil is kept at a temperature of 15 to 20 °C for a period of 24 hours and then examined for the presence of sediment and for other insoluble matter. See also 5.11, Foots.

5.4 Refractive index

Refer to the Annex A, subject to the provision that ISO recommends taking the reading at 20 °C (and if necessary at 40 °C in countries with a tropical climate).

5.5 Volatile matter

Weigh about 10 g of oil (m g) with an accuracy of 1 mg into a 200 ml conical flask (diameter of the bottom 73 mm, height 125 mm). Heat the flask for 30 min in an oil bath of 105 to 110 °C, drawing a dry inert gas over the surface of the oil at a rate of 10 litres per hour. Remove the inert gas before weighing. (Loss of mass m_1 g.)

Calculate the percentage of volatile matter (V) with the formula:

$$V = \frac{m_1}{m} \times 100$$

in which

m = mass, in grammes, of the sample
m_1 = loss of mass, in grammes.

5.6 Ash

5.7 Acid value

5.8 Saponification value

Refer to the Annex A.

5.9 Unsaponifiable matter

Refer to the Annex A.

The solvent to be used should be agreed upon between purchaser and vendor.

5.10 Iodine value

Refer to the Annex A.

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* At present Second Draft ISO Recommendation No. 731.

5.11 Foots

5.11.1 *Apparatus.* The test is carried out in a cone-shaped or a pear-shaped sediment tube (see Fig. 1 and 2 below).

5.11.2 *Procedure.* Place 100 ml of the oil in the tube and allow it to stand there in a vertical position for 4 days (96 hours) at a temperature between 15 and 20 °C. During this time, the tube should remain undisturbed. At the end of the 4 days (96 hours), read the volume of sediments or foots at the bottom of the tube. Report that value as the percentage of foots, by volume.

Dimensions in millimetres

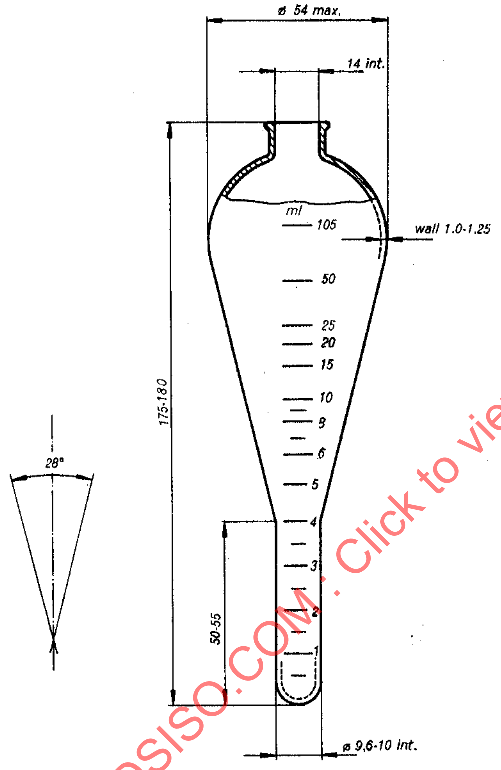

FIG. 1.— **Pear-shaped sediment tube**

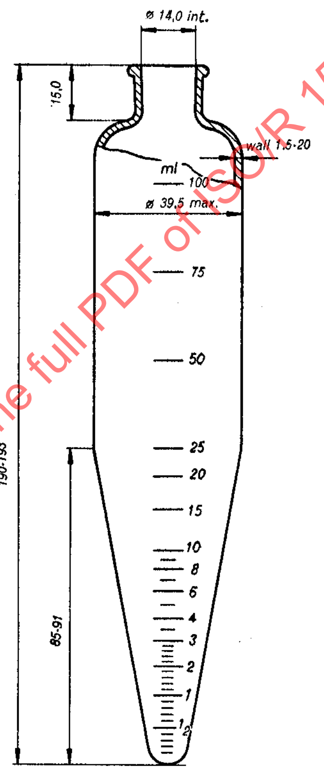

FIG. 2.— **Cone-shaped sediment tube**

5.12 Drying time

Spread two drops of oil (about 75 mg) with the finger evenly over the whole surface of a glass panel, dimensions 9 cm × 12 cm.

Place the panel in diffuse daylight in a room at a temperature between 15 and 20 °C and at a relative humidity between 60 and 70%.

The end of the drying time is when clean sand, graded so as to pass a sieve with a width of aperture of 0.315 mm, but to be retained on a sieve with a width of aperture of 0.160 mm, placed on the surface of the panel and allowed to remain there for a minute, can be removed by means of a camel-hair brush without injury to the oil film.

5.13 Liebermann-Storch-Morawski test for presence of colophony

Mix 3 to 5 drops of the oil with 3 ml of acetic anhydride. Add 1 drop of sulphuric acid ($d = 1.54$). The reaction for colophony is positive if a fugitive violet colour appears which, at the moment of its maximum intensity, is stronger than the colour of potassium permanganate solution 0.001 N.

5.14 Test for presence of fish oil

5.14.1 *Reagents*

5.14.1.1 Acetic acid, glacial.

5.14.1.2 Nitric acid 4 N.

5.14.1.3 Hydrochloric acid 1 N.

5.14.1.4 Ethanolic potassium hydroxide solution 1 N.

5.14.1.5 Mixture of equal volumes of acetic acid and chloroform.

5.14.1.6 Bromine, analytical grade.

5.14.1.7 Chloroform.

5.14.1.8 Light petroleum (boiling range 40 to 60 °C).

5.14.1.9 Diethyl ether, $d = 0.712$ to 0.716, the residue of non-volatile matter at 80 °C not exceeding 0.001 %.

5.14.2 *Procedure*

Saponify 5 g of linseed oil, as in the determination of unsaponifiable matter (see clause 5.9), using 50 ml of the ethanolic potassium hydroxide solution and boiling under reflux for one hour, with swirling at intervals.

Remove the flask from the condenser and evaporate the contents to a syrupy paste on a water or steam bath.

To ensure removal of ethanol, add 10 ml of water and again evaporate, almost to dryness.

Dissolve the soap in about 100 ml of boiling distilled water and transfer to a separating funnel. Add an excess of hydrochloric acid and shake vigorously to decompose the soap. Cool and add about 200 ml of light petroleum. Shake well and allow to stand for several hours. Run off the acid layer. Filter the light petroleum layer through a fast-filtering, fat-free paper, pouring the liquid from the top of the separating funnel. Wash the filtered solution with 10 ml of nitric acid. Allow to settle, run off the acid layer and wash several times with distilled water. Repeat the acid and subsequent water washes until no trace of metal is present. Evaporate the light petroleum and carefully dry the residual fatty acids.

Take 1.5 ml of the residual fatty acids and dissolve in 40 ml of diethyl ether and 5 ml of glacial acetic acid in a 25 mm × 150 mm test tube. Cool the solution in an ice-water bath to 0 °C.

Add bromine drop by drop, keeping the temperature at 0 °C, until an excess, as indicated by the colour, is present. Allow to stand at 0 °C, preferably overnight. Decant the supernatant diethyl ether-bromine mixture and wash the precipitated polybromides until they are free from bromine into a filter paper with diethyl ether previously cooled to about 0 °C. Allow the ether to evaporate from the bromides.

Take 0.1 to 0.2 g of the dry bromides thus prepared and add acetic acid-chloroform mixture in the proportion of 1.5 ml of mixture to 0.1 g of bromides and boil.

Vegetable oils give a clear solution. When 1 % of fish or marine animal oil is present, the solution does not become bright; when 5 % is present, the mixture is quite cloudy.

5.15 Test for presence of mineral acid

Shake 10 ml of refined linseed oil with 25 ml of distilled water. Take off the aqueous layer and add a few drops of litmus solution.

In the presence of mineral acid the colour turns to red.

ANNEX A

(prepared in 1966)

Extracts from the publications of the

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY (IUPAC)

Standard methods of the

OILS AND FATS SECTION (OFS)

and

ORGANIC COATINGS SECTION (OCS)

A.1 DENSITY

A.1.1 General

In view of the confusion which may be caused by the terms *density* and *specific mass* in the different countries, and sometimes even in one country, the term *density at t* °C is defined as the absolute density, that is to say, *the mass of unit volume*, expressed in grammes per millilitre at the temperature indicated t. This quantity is indicated by the symbol ρ_t.

To within 0.000 027, that is to say, within a degree of approximation closer than that obtained in ordinarily accurate measurements, this quantity will correspond with the density in relation to water at 4 °C: ρ_4.

The exact determination of the temperature t is of considerable importance since the density of the fat varies by about 0.000 68 per degree C.

Densities, like refractive indices, shall be determined on the dried and filtered fat and shall be quoted at 20 °C (and if necessary at 40 °C, 60 °C, or other temperatures, if the substance does not have a well defined state at 20 °C).

The observations may be made at a temperature t_1, approximating to the standard temperature t, and be corrected to the latter temperature by means of the equation:

$$\rho_t = \rho_{t_1} + (t_1 - t) \times 0.000\,68$$

A.1.2 Procedure

The density bottle, having reached the constant temperature of the surrounding medium, is filled to the upper edge of the capillary tube. After the thermometer has been inserted, the density bottle is weighed, the temperature being noted.

The density at t °C (ρ_t) is then calculated by the following formula:

$$\rho_t = \frac{c - a}{b - a} \Delta$$

in which
a = weight of empty density bottle,
b = weight of density bottle plus water at t °C,
c = weight of density bottle plus oil at t °C,
Δ = density of water at t °C (to be ascertained from tables).

The approximate value of ρ_t determined as above may now be corrected for the effect of the air pressure by means of the following formula:

if (ρ_t) is the corrected value and 0.0012 the density of air
then $(\rho_t) = \rho_t + 0.0012\,(1 - \rho_t)$.

A.2 REFRACTIVE INDEX

A.2.1 Foreword

The refractive index of a medium is the ratio of the speed of light *in vacuo* to its speed in the medium. It is measured by the ratio of the sine of the angle of incidence to the sine of the angle of refraction when a ray of light passes from air into the oil or fat. In general, the refractive index of a given substance varies with the wave length of the refracted light and with the temperature.

Unless otherwise specified, the refractive index is reported for the mean wave length of the D lines of sodium.

The notation n^t always denotes the index relative to the D line at t °C.

For a light of wave length x at t °C, the notation is n_x^t.

In the case of refractometers in common use, the observation is made in white light but is corrected to sodium light by a compensating device in the instrument.

In general, the refractive index is given

at $t = 20$ °C for fats which are liquid at this temperature;
at $t = 40$ °C, 60 °C, 80 °C or above for fats which are solid at a temperature of 20 °C.

The temperature of observation t_1 must be accurately known and should not differ from the selected temperature by more than ± 2 °C.

A.2.2 Apparatus

A precision refractometer having a prism heated by means of a thermostatically controlled circulating liquid. An instrument reading only in arbitrary units is not regarded as suitable.

A.2.3 Measurement

The space between the two prisms must be completely filled with the fat.

The reading is taken after the temperature has remained constant for not less than 5 minutes.

Note the temperature, say t_1.

A.2.4 Calculation

If t_1 is different from t, the correction factor F is a function of the temperature:

$F = 0.000\,35$ for temperatures around 20 °C.
$F = 0.000\,36$ for a temperature of 40 °C and above.

We have then

$$n^t = n^{t1} + (t_1 - t)F \text{ when } t_1 > t$$
$$n^t = n^{t1} - (t - t_1)F \text{ when } t_1 < t$$

A.3 ASH CONTENT

A.3.1 Foreword

The ash content is the percentage of mineral matter found in oils and fats which have been filtered.

In order to avoid the inclusion of certain elements twice the ash content should not be added to that of the impurities insoluble in a solvent.

On the other hand, the ash can be determined on the purified matter when the determination is being made after that of the impurities.

A.3.2 Apparatus

A 50 ml crucible.

A.3.3 Procedure

Weigh about 10 g of the fatty material to within 0.01 g in the previously tared crucible.

Heat gently to point of ignition and allow to burn spontaneously. Ignite until a carbon residue is obtained which no longer emits a vapour.

Take up the residue with distilled water. Filter through an ashless filter paper. Keep the filtrate. Ignite the filter completely, if necessary with the help of a gentle stream of oxygen or a few drops of hydrogen peroxide.

Allow the crucible to cool.

Pour the filtrate quantitatively into it. Evaporate to dryness over a water-bath. Then ignite for a few moments at about 400 °C.

Finally recarbonate the ash with ammonium carbonate or water saturated with carbon dioxide.

A.3.4 Calculation

$$\text{Ash content} = \frac{100a}{p} \text{ per cent}$$

where

a = weight of ash in grammes
p = weight of sample in grammes.

A.4 ACID VALUE *

A.4.1 Definition

The acid value is defined as the number of milligrammes of potassium hydroxide required to neutralize 1 g of the sample under the conditions specified below.

A.4.2 Reagents required

Standard potassium hydroxide solution (0.1 N) in ethanol (95% v/v). This solution must be colourless or not darker than straw yellow. Methanol may be used instead of ethanol (95% v/v).

1 % v/v of phenolphthalein in ethanol (95% v/v). Other indicators with a colour change in the same pH-range may be used also.

Ethanol-toluene mixture. Mix equal volumes of ethanol (95% v/v) and toluene and neutralize this mixture with ethanolic potassium hydroxide solution (0.1 N) in the presence of phenolphthalein as indicator.

A.4.3 Procedure

Weigh to the nearest 0.01 g into a conical flask, 2 to 10 g of the sample, according to the expected acid value.

Add 50 ml of ethanol-toluene mixture to the sample and shake until solution is complete.

Titrate with ethanolic potassium hydroxide solution in the presence of phenolphthalein as indicator.

A.4.4 Calculation

Calculate the acid value as follows:

$$\text{Acid value} = 56.1\, aN/p$$

where a = ml ethanolic potassium hydroxide solution,
N = normality of the ethanolic potassium hydroxide solution,
p = weight of the sample in grammes.

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* The solvent used in this method differs from that prescribed in the corresponding method of the "Standard Methods of the Oils and Fats Section of the IUPAC", because the ethanol-toluene mixture is sable for polymerized as well as for unpolymerized drying oils.

A.5 SAPONIFICATION VALUE

A.5.1 Definition

The saponification value is defined as the number of milligrammes of potassium hydroxide required for the saponification of 1 g of the sample under the conditions specified below.

A.5.2 Apparatus

Conical flasks of alkali-resistant glass, 200 or 300 ml capacity.
Reflux condensers. The use of interchangeable ground-glass joints is recommended.

A.5.3 Reagents required

Standard hydrochloric acid solution (0.5 N).

Potassium hydroxide solution (0.5 N) in ethanol (95% v/v). This solution must be colourless or not darker than straw yellow.

1% v/v of phenolphthalein in ethanol (95% v/v). Other indicators with a colour change in the same pH-range may be used also.

A.5.4 Procedure

Weigh to the nearest 0.001 g into a conical flask, approximately 2 g of the sample.

Add exactly 25 ml of ethanolic potassium hydroxide solution to the sample by means of a burette or another suitable instrument of equal precision, connect the flask with a condenser and reflux for 1 hour, shaking from time to time.

Titrate the soap solution, while still warm, with hydrochloric acid solution in the presence of phenolphthalein as indicator.

Carry out a blank by refluxing and titrating exactly 25 ml of ethanolic potassium hydroxide solution under the same conditions.

A.5.5 Calculation

Calculate the saponification value as follows:

$$\text{Saponification value} = 56.1\,(b - a)\,\mathrm{N}/p$$

where a = ml hydrochloric acid required for the titration of the sample,
b = ml hydrochloric acid required for the titration of the blank,
N = normality of the hydrochloric acid solution,
p = weight of the sample in grammes.

NOTE. — The saponification value as determined is a measure of the sum of the free and hydrolysable acids.

A.6 UNSAPONIFIABLE MATTER *

A.6.1 General

By unsaponifiable matter of oils is meant the substances soluble in oils which, after saponification, are insoluble in water and soluble in the solvent used for the determination.

It includes the naturally occurring unsaponifiable substances (*e.g.*, sterols, alcohols, hydrocarbons) as well as the foreign organic unsaponifiable substances (*e.g.*, mineral oils) which may have been added to the oils.

By the methods described below, only the unsaponifiable substances not volatile at 100 °C are determined.

Petroleum ether or ethyl ether is used as solvent. One has to be aware of the fact that the results obtained with the two solvents are in most cases different, the results with ethyl ether usually being higher than those with petroleum ether.

A.6.2 Petroleum ether method

A.6.2.1 *Apparatus*

Round-bottomed flasks of about 250 ml capacity.

Reflux condenser. The use of interchangeable ground-glass joints is recommended.

Separating funnels, 500 ml capacity.

Drying oven adjusted at 103 ± 2 °C.

A.6.2.2 *Reagents required*

Standard potassium hydroxide solution (0.1 N) in ethanol (95% v/v).

Potassium hydroxide solution (2 N) in ethanol (95% v/v). This solution must be colourless or not darker than straw yellow.

Solution of potassium hydroxide (0.5 N) in ethanol (50% v/v).

Nitric acid. Mix one part of concentrated acid ($d = 1.40$) with three parts of water.

1 % v/v of phenolphthalein in ethanol (95% v/v).

Ethanol (95% v/v), neutralized.

Ethanol (approximately 50% v/v). Mix equal volumes of ethanol (95% v/v) and water.

Petroleum ether (boiling range: 40 to 60 °C), free from residue, bromine index less than one.

Acetone (pure).

A.6.2.3 *Procedure*

Weigh to the nearest 0.01 g into a round-bottomed flask about 5 g of the sample, previously warmed and filtered if necessary.

Add 50 ml of ethanolic potassium hydroxide solution (2 N), connect the flask with a reflux condenser and reflux the mixture gently for one hour, shaking from time to time.

Remove the condenser from the flask and transfer the contents of the flask to a separating funnel. Rinse the flask several times with a total of 50 ml of distilled water and pour the water into the separating funnel.

Rinse the flask several times with a total of 50 ml of petroleum ether and pour the petroleum ether also into the separating funnel.

Allow to cool to between 20 and 25 °C, stopper the funnel, shake vigorously for one minute and allow to stand until the two layers have clearly separated.

Run off the soap solution into the flask used for the saponification.

Pour the petroleum ether solution through the top of the funnel into a second separating funnel containing 40 ml of water.

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* In view of the possible presence of metal soaps in the oil, as, *e.g.*, in boiled linseed oil, the above methods include a washing with nitric acid. In this respect the methods differ from those described in the " Standard Methods of the Oils and Fats Section of the IUPAC". In the absence of metal soaps, the washing with nitric acid may be omitted.

Extract the soap solution in the same manner twice more, each time with 50 ml of petroleum ether, using the first separating funnel, and combine the three extracts in the second separating funnel.

When resistant emulsions are formed, they may be broken down by the addition of a small quantity of ethanol or methanol. When the extracts contain any suspended matter, filter them and wash the filter with a little petroleum ether.

Rotate the funnel containing the combined extracts and the 40 ml of water without violent shaking, and, after it has separated, run off the water.

Wash the petroleum ether solution by shaking vigorously

twice with 50 ml of ethanol (50% v/v),
once with 10 ml of nitric acid,*
once with 50 ml of ethanol (50% v/v),*
once with 50 ml of potassium hydroxide (0.5 N) in ethanol (50% v/v),
once with 50 ml of ethanol (50% v/v).

Repeat twice the last two washings and wash with ethanol (50% v/v) until neutral on phenolphthalein.

Transfer the petroleum ether solution—if necessary in several portions—through the top of the funnel quantitatively into a weighed round-bottomed flask, rinsing the funnel with small amounts of petroleum ether.

Evaporate to a small volume by distillation on a water-bath, add 6 ml of acetone and evaporate the solvent completely by means of a gentle stream of air while the flask is held obliquely and almost submerged in a boiling water-bath and rotated.

Dry the residue by placing the flask for 15 minutes in a horizontal position in a drying oven adjusted at 103 °C.

Allow the flask with its contents to cool in a desiccator and weigh.

Repeat the drying for successive periods of 15 minutes until the loss between two weighings is less than 0.5 mg.

Dissolve the residue in 20 ml of ethanol (95% v/v) and titrate the solution with ethanolic potassium hydroxide (0.1 N) in the presence of phenolphthalein as indicator.

If more than 0.2 ml of potassium hydroxide solution is used, the determination must be completely repeated, giving more attention to the washings.

A.6.2.4 *Calculation*

Calculate the contents of unsaponifiable matter as follows:

$$\text{Unsaponifiable matter (per cent)} = a/p \times 100$$

where a = weight of the residue in grammes,
p = weight of the sample in grammes.

Give the results to the first decimal place and indicate whether the petroleum ether method or the ethyl ether method is used.

A.6.3 Ethyl ether method

A.6.3.1 *Apparatus*

See " Petroleum ether method ".

A.6.3.2 *Reagents required*

Standard potassium hydroxide solution (0.1 N) in ethanol (95% v/v).
Potassium hydroxide solution (2 N) in ethanol (95% v/v). This solution must be colourless or not darker than straw yellow.
Potassium hydroxide solution (0.5 N).
Nitric acid. Mix one part of concentrated acid (d = 1.40) with three parts of water.
1 % v/v of phenolphthalein in ethanol (95% v/v).
Ethanol (95% v/v), neutralized.
Ethyl ether free from residue.
Acetone (pure).

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* In the absence of metal soaps, this washing may be omitted.